



## **Nordic collaboration on the use of mass-spectrometers for the analysis of radioisotopes. NKS-project NORCMASS. Final report**

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# Nordic Collaboration on the use of Mass-Spectrometers for the Analysis of Radioisotopes. NKS-project NORCMAS

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## Abstract

This report cover an overview of the work performed during a three year (2003-2005) project initialized with the purpose of identifying and work on problems in isotope ratio and ultra trace measurements of primarily plutonium and uranium isotopes and  $^{237}\text{Np}$  using ICP-MS. The project also included an educational part aiming to describe fundamental aspects and practical steps for radioisotope measurements using ICP-MS.

## Key words

Norcmass, ICP-MS, Mass spectrometry

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## **NKS-project NORCMASS.**

### **Nordic Collaboration on the use of Mass-Spectrometers for the Analysis of Radioisotopes.**

#### **Final report**

This report covers the work performed during a three year (2003-2005) project initialized with the purpose of identifying and work on problems in isotope ratio and ultra trace measurements of primarily plutonium and uranium isotopes and  $^{237}\text{Np}$  using ICP-MS. The project also included an educational part aiming to describe fundamental aspects and practical steps for radioisotope measurements using ICP-MS.

The project was separated into the 12 following stages.

1. An initial workshop to identify & suggest solutions to existing problems within high precision isotope ratio measurements (of U and Pu) and analysis of ultra low levels of long lived radioisotopes performed on single collector instruments.
2. Follow up studies on relevant problems identified as necessary under P1
3. Production of a Nordic isotope ratio reference material (for  $^{240}\text{Pu}/^{239}\text{Pu}$  and  $^{234}\text{U}/^{235}\text{U}/^{238}\text{U}$ ).
4. Including measurements of  $^{237}\text{Np}$  in the Nordic isotope reference material due to interest in  $^{237}\text{Np}/^{239}\text{Pu}$  ratios.
5. A workshop on radioisotope measurements with ICP-MS (new members) in Örebro 23/6-2004.
6. Workshop 18/8-2004 on ultra low measurements, isotope ratios & necessary radiochemistry at Risoe, Denmark (partly in co-operation with the NKS-B RADCHEM project).
7. Presentation of project work at the NKS-B seminar at Risoe in 18/8-2004.
8. Second workshop on ultra low measurements, isotope ratios & radiochemistry. Helsinki 17-18/2 2005 (in co-operation with the NKS-B RADCHEM project).
9. Presentation of work conducted within Norcmass at the NKS-B seminar in Tartu, Estland 24-25/10 2005.
10. Production of a "Guideline Book" aimed for beginners in the field of radioisotope measurements using ICP-MS.
11. Planning of a practical training course in isotope ratio measurements
12. Writing a summary of conclusions and recommendations.

## **(1) Initial workshop**

The workshop to identify & suggest solutions to problems with ultra low measurements and high precision ratio measurements of Pu and U isotopes was formally held at Umeå (FOI) on the 22 October although the problems around such measurements has been discussed within the group since the start of the project. It has earlier, and was further discussed at the workshop, been decided to put more strenght on the mesurements of the short time signal variations (single ion monitoring) as a function of nebuliser selection and plasma conditions. Examples of measured short (ms) signal variations (Risö) was shown and discussed. An evaluation on differences in isotope ratio precision from different types of sample introduction systems should be useful. The standard sample introduction system delivers the sample in a wet condition to the plasma which is known to be unfavorable from a sensitivity point of view but which also should be unfavorable in isotope ratio measurements. It was decided that the effect of desolvating systems should be investigated. Also other types of fluctuations connected to this part of the instrument, such as pump versus self-aspiration and gas flows should be tested. A different, but highly relevant issue, which was brought up was the present difficulty in obtaining certified reference material for plutonium isotopes from suppliers both in Europe and the US.

Apart from these issues a large number of specific problems of technical and operational nature around the use of mass spectrometry and associated radiochemistry for radioisotope analysis were also discussed. About half of the meeting was devoted to running the MS situated at FOI for the purpose of comparing the response and behavior (on reference materials) relative other instruments in the group. In general the meeting was very fruitful for all parts and it was decided that similar measurement-workshops must be held more often.

## **(2) Follow-up on problems identified under P1 above**

During the present project work has been started to analyse factors which influence the short signal variations which is operating at the same time scale as the peak hopping frequency. A comparison between cyclonic and ultrasonic nebulisers have also been initiated in order to test if a difference in short time signal stability (relevant for isotope ratio precision) exist for wet and dry aerosol delivery to the plasma. Work has also been conducted to produce a radiochemical procedure for measuring ultra trace amounts of plutonium where in principal nearly all uranium must be removed.

## **(3+4) Production of a Nordic isotope ratio reference material for $^{240}\text{Pu}/^{239}\text{Pu}$ and $^{234}\text{U}/^{235}\text{U}/^{238}\text{U}$ as well as for $^{237}\text{Np}$ .**

This is given as a separate report.

## **(5) A workshop on radioisotope measurements with ICP-MS (new members) in Örebro 23/6-2004. Mini-seminar**

The seminar on radioisotope measurements was held at Örebro on the 23/6-2004. The new members (Univ. of Örebro and Univ. of Linköping) were introduced and experiences were shared. A tour of the Örebro facilities were given showing the quadrupol instrument situated in a clean-room equipped with a USN+desolvating system. The main part of the meeting dwelled upon technical details around the sample introduction system and difficulties in correcting for remaining uranium in the samples when performing Pu-isotopic measurements. The main

interest for the Örebro/Linköping group is to perform rapid methods for Pu-analysis using ICP-MS. It seems as if one of the main obstacles in performing reliable measurements still is to be found in the separation chemistry and it was decided that it might be useful to discuss the problems with the existing NKS-B project 'RADCHEM'. The meeting agenda is found in appendix 1.

#### **(6) Workshop on ultralow measurements, isotope ratios & necessary radiochemistry**

The workshop was held partly together with the NKS-B RADCHEM project at Risø on the 18 of August 2004. An important conclusion of the meeting was that there are very large differences in knowledge among the participating members on how to actually perform precise low level measurements of Pu & U isotopes. Such differences are first seen when going into details of the analytical procedures coupling radiochemistry to ICP-MS. Observed difficulties are partly due to a tradition in some laboratories to separate the radiochemical work from the instrument work (different persons doing the tasks) which results in a lack of insight in what is needed or not (e.g. influence of polyatomic interferences and therefore the need to remove certain elements like lead and mercury). It also became evident that the actual number of persons capable of operating ICP-MS instruments and correctly performing low level Pu & U isotopic measurements (including the necessary chemical separation) are surprisingly low – amounting to less than 3-4 persons in the Nordic countries. Meeting agenda as appendix 2.

#### **(7) Presentation of project work at the NKS-B seminar at Risø 18/8-2004.**

This was done according to plan. Extended abstract for one of the participants (Risø) given as appendix 3.

#### **(8) Second workshop on ultra low measurements, isotope ratios & radiochemistry. Helsinki 17-18/2 2005 (in co-operation with the NKS-B RADCHEM project).**

Presentations of work performed at the laboratories. FOI-Umeå presented interesting studies, which shows that there are factors outside the normal instability of the plasma and sample introduction system that influence the precision of isotope ratios. In this particular case the software, which by pooling individual data causes a truncation induced bias. A manuscript intended for Journal of Analytical Atomic Spectrometry had been submitted.

Contact was made to students at the Laboratory for Radiochemistry who had interest in performing measurements using ICP-MS and therefore had potential interest in the training course set up in this project.

#### **(9) Presentation of work conducted within Norcmass at the NKS-B seminar in Tartu, Estland 24-25/10 2005.**

Part of work performed during the three year project at two of the laboratories (Risø and FOI-Umeå) were presented.

**(10) Production of a “Guideline Book” aimed for beginners in the field of radioisotope measurements using ICP-MS.**

This has been an ongoing task along the project period. The Guideline Book covers the basic functioning in ICP-MS and the practical aspects when working with the instrumentation as well as some guidance on how to correct or avoid interfering signals and how to analyse low levels of Pu and U by ICP-MS. The Guideline Book is given as a separate report.

**(11) Plan a practical training course in isotope ratio measurements**

A training course aimed to be used at the Risoe sector field ICP-MS has been drawn up. The schedule is given together with the Guideline Book.

**(12) Summary of conclusions and recommendations.**

The following is a brief summary of the most essential findings done during the project period with respect to measurements of isotope ratios and/or ultra low concentrations of Pu and U.

It should be emphasized that the main advantage with the project has not been the scientific findings produced during the project but merely the chance to bring together scientists interested in field. The discussions during meetings, e-mail and telephone have stimulated the thoughts around isotope- and ultra low measurements in a way that would not have been possible without this project. Certainly, the skill among the participants to perform such measurements has improved due to the project. This is perhaps also the most significant outcome of the project.

Experiences tells us that without any radiochemical treatment of the sample practical limits of Pu isotopic measurements are at the order of kBq with present quadrupole based ICP-MS instruments. For sector field ICP-MS instruments limits are about 1-3 orders of magnitude better if high resolution modes are used. The limitations are due to abundance sensitivity of the instruments. If reliable measurements are to be performed at lower levels (either concentrations or ratios) radiochemical treatment is unavoidable and becomes more critical the lower the concentrations. Clearly, the limitations in precision in isotope ratio measurements as well as detection limits are not set by the instruments but rather by the operators. The specific coupling of separation chemistry and instrument knowledge which characterize these type of measurements requires substantial training and experience. With training the separations may match the limits of the instruments (around femtograms). The measurements of Pu or Np at the sub pg levels are far from trivial and much more complicated than what is normally imagined. Reliable results require skilled staff capable of recognizing critical steps both with respect to the instrument set-up and to the radiochemistry involved. Recommendations are therefore that the experimental training in the field is supported at all levels. In order to take advantage of existing equipment time must be allocated to allow for testing both with respect to the separation chemistry involved and, in particular, to allow time to be used for experimenting on the response of the instruments using different kind of separation chemistry.

Interferences, contribution due to poor abundance sensitivity, matrix suppression of signal and mass fractionation all depend on the quality of sample separation chemistry and is thus essential. As a starting point the same chemical separation procedures, which is used for

radiometric measurements, may be used for samples intended for ICP-MS but a final purification step is necessary. This step must include both good removal of uranium as well as some of the heavy metals which may cause polyatomic interferences with the Pu and U isotopes. In order to minimize the contribution from the chemicals themselves it is important to minimize the volumes of chemicals. For ion-exchange columns this typically means sub-ml columns.

Instrument specific settings also influence the precision and those are mainly the way in which the measurement protocol is set up. Rapid peak hopping between the central part of flat-topped peaks instead of peak scanning tend to provide better precision (and also results in better counting statistics). This peak hopping is also possible with sector field instruments since the low mass range (234-242 for Pu and U isotopes) may be performed by voltage settings in the flight tube and thus not need to involve the magnet. Theoretically, the measurement protocol could be designed from the analysis of the short time intensity fluctuations observed for each sample. In practice however this is not easily accomplished due to fixed settings in most software used for the measurement.

## **Reports**

Although much work has been performed linked to the project far from all has been evaluated and reported. We here only include reported work.

### **List of reports**

- *Pu-isotope measurements at femtogram levels using sector field ICP-MS*. Per Roos, Risoe National Laboratory. Presented at the International Conference on Isotopes in the Environmental studies – Aquatic forum. Monaco 25-29 October 2004. In conference proceedings.
- *Pu-isotope measurements at femtogram levels using sector field ICP-MS*. Per Roos. Manuscript accepted for special issue JER.
- *Comparison of different sample inlet systems for analysing Pu-isotopes*. Lindis Skipperud, Agricultural University of Norway. Unpublished work 2004.
- Rapid Method for ICP-MS Analysis of Plutonium in Sediment Samples. Christina Greis, Anders Duker, Håkan Pettersson, Stefan Karlsson and Bert Allard (Univ. of Örebro and Univ. of Linköping). In: Stroes-Gascoyne, S., Hanchar, J., Browning, L. (Eds.) *Scientific Basis for Nuclear Waste Management XXVIII*. Materials Research Society, Warrendale. vol 824.
- Transuranium elements and their analyses in environmental samples – a literature review: Man-Technology-Environment Research Centre, Örebro University. MTM 04-5 PP. C. Greis (2004)
- Greis, C., Düker, A., & Karlsson, S. (2004). *Sources of plutonium in the environment and rapid methods for determination as emergency measures*. Paper presented at the 227th ACS National Meeting (poster), Anaheim



- Greis, C., & Allard, B. (2005). Analysis of actinides in environmental matrices by icp-ms – a procedure based on chemical speciation: Man-Technology-Environment Research Centre, Örebro University.
- Anna Sjögren, Petra Appelblad, Annika Tovedal and Henrik Ramebäck. Journal of Analytical Atomic Spectrometry, 20, 320-322, (2005). Isotope amount ratio measurements by ICP-MS: aspects of software induced measurement bias and non-linearity.
- Ulrika Nygren, Henrik Ramebäck, Douglas Baxter and Calle Nilsson. Journal of Analytical Atomic Spectrometry, 20, 1-6, (2005). Lanthanide phosphate interferences in actinide determination using inductively coupled plasma mass spectrometry.

## **Appendix 1**

### **NKS-Norcmass möte, Örebro 23/6-2004**

#### **Preliminära punkter för mötet:**

- Allmän projektbakgrund.
- Kontraktet med NKS – individuella punkter (se kontraktet).
- Fördelning av arbetsuppgifter
- Eventuella presentationer.
- Tekniska diskussioner om analys av långlivade radioisotoper.
- Referensmaterial/tracers – gemensamma inköp?
- Möte i Augusti.
- Visning av laboratorier

## **Appendix 2**

### **NKS-Norcmass möte, Risö 18/8-2004**

#### **Preliminära punkter för mötet:**

- Presentation av utförda experiment.
- Kontraktet med NKS – individuella punkter (se kontraktet). Var är vi?
- Fördelning av arbetsuppgifter – Guideline book.
- Training course – var, när & hur.
- Tekniska diskussioner om analys av långlivade radioisotoper –  $^{237}\text{Np}$ ?
- Samarbete med NKS-Radchem gruppen.
- Referensmaterial/tracers – gemensamma inköp?
- Visning av laboratorier
- Eventuellt.

**Isotope ratio measurements with single collector ICP-MS**

Per Roos, Risoe National Laboratory

The ICP-MS technique has during the last 20 years become increasingly important in the analysis of long-lived radioisotopes. Although useful both with respect to rapid measurements as well as performing highly accurate isotope ratio determinations and analysis of extremely low levels the technique requires careful attention. The need for routine use in producing data often overshadows the need to look into the many parameters governing the quality of data. This is particularly important in the analysis of long lived radioisotopes due to the often extremely low concentrations and/or the need to perform isotopic ratio determinations with high accuracy of extreme ratios (U&Th isotopes).

When considering the need to measure isotope ratios of Pu and U isotopes in environmental samples there are thus a number of issues that deserves investigation in order to ensure quality of the data. These could roughly be grouped as follows:

- Sample introduction system
- Plasma instability
- Dead time corrections
- Mass fractionation
- Interferences
- Abundance sensitivity (peak tailing)

**Sample introduction system**

One of the main advantages with the ICP-MS technique which was early emphasized was the possibility to introduce the aqueous sample directly into the instrument without any pre-treatment. This was a significant step forward compared to for instance the sample introduction used in thermal ionization mass spectrometry (TIMS) where samples has to be loaded onto thin filaments after a very tedious chemical refinement of the sample atoms. However, whereas the TIMS sample just need heating (which may be controlled with high accuracy) to produce an ion beam the ICP-MS requires the aqueous sample to pass through a complex chain of operations in order to produce the ion beam to the mass spectrometer. This chain of events causes a much greater fluctuation in the ion beam intensity than what is seen in the TIMS signal. The sample introduction system in ICP-MS may be of very different types but for aqueous samples they all have the task of delivering the sample in a form which makes the plasma being able to efficiently transform it into a an ion beam. This often means producing an aerosol (nebulization) which in turn may be dried from water and thereafter introduced into the plasma where the sample is atomized and ionized. Since the amount of sample introduced to the nebuliser depends on pump movements and gas flows, signal fluctuations are present already at this point. The sample loading further influences the aerosol production both with respect to magnitude and aerosol size distribution, both parameters which influences the signal intensity due to variations in aerosol transport and in residence time in the plasma. In reality it is very difficult to remove the signal variations caused by the sample introduction system. Although the pump movements may be omitted by allowing for self-aspiration of the sample

this noise are often of low frequency and thus plays a minor role in the precision of isotope ratio measurements provided the mass scanning is not in phase with the noise. Another way of minimising the effect of the sample introduction system is to minimise the sample input rate and to use desolvating equipment to remove all water from the aerosol stream before reaching the plasma. Since measurements of radioisotopes at environmental levels often are preceded by a radiochemical separation process there is a natural way of keeping the sample small (sub millilitre) and thus partly avoiding the sample introduction problems.

### Plasma instability

One of the main problems with isotope ratio measurements in ICP-MS is the plasma instability. The plasma flickering causes a continuous signal variation due to variation in ionisation/atomisation efficiency and to variations in ion kinetic energy. Although there are no simple ways of avoiding the plasma flickering it may be reduced by the use of a shielded torch. Another way of reducing the influence of the plasma flickering is to optimise the mass scanning frequency with respect to the plasma flickering frequency so they are not in phase. This is often practically easy since the plasma flickering may show a rather continuous frequency distribution. Fast scanning usually however show better precision in isotopic ratios than slow scanning but this may also be due to avoiding the noise from the sample introduction system.

### Dead time corrections

A factor sometimes of importance is the corrections for dead time. This is usually not necessary when considering Pu-isotopes in environmental samples but may certainly be important when measuring U and Th isotope ratios ( $^{235}\text{U}/^{238}\text{U}$ ,  $^{234}\text{U}/^{238}\text{U}$ ,  $^{236}\text{U}/^{238}\text{U}$  or  $^{230}\text{Th}/^{232}\text{Th}$ ) where the ratios may be extremely large. In such cases the ion beam intensity for the major isotope may reach millions of counts per second while it may be less than 1cps for the minor one. The methods frequently used to correct for the dead time may be either as in radiometric counting (observing the ratio of measured to introduced sample as a function of sample introduction rate) or by using samples having certified isotope ratios of the selected isotopes and using them in the same concentration as the actual sample(s). There are a number of well suited certified reference samples available. Figure 1 shows the frequently used U500 from NBL having a  $^{235}\text{U}/^{238}\text{U}$  ratio of 1 and which also contains the low abundance isotopes  $^{234}\text{U}$  and  $^{236}\text{U}$ .

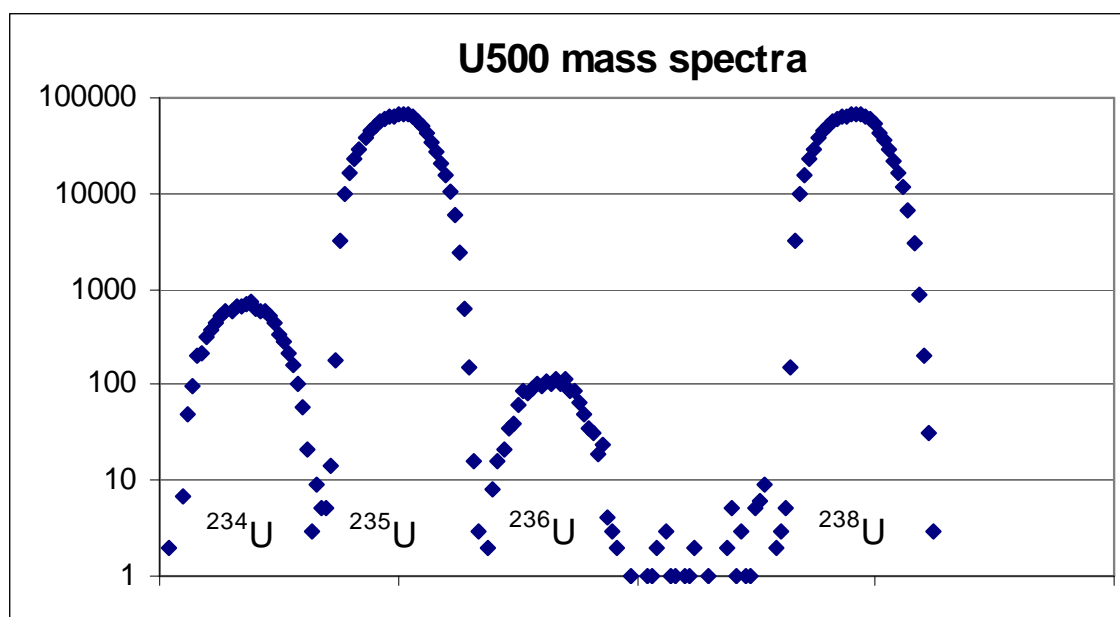


Figure 1: Typical mass spectrum of the certified reference material NBL U500. Obtained in low-resolution mode ( $R=300$ ). PlasmaTrace 2 HR-ICP-MS, Risö National Laboratory.

### Mass fractionation

Mass fractionation may occur between isotopes of different mass due to their different ability to be transported through the MS optics. Typically transmission of higher mass isotopes is somewhat larger than the lighter ones due to the fact that the lighter ones are more easily repelled away from the ion beam, especially in high intensity beams (samples with high concentration of the analyte or with a high salt content). The correction for mass fractionation is obviously a small problem for the heavy actinides since the relative difference in mass is rather small. Nevertheless, corrections may be necessary in high precision work and may be performed with the abovementioned U500 reference material with well known isotope ratios, provided the measurements are done at low count rates to minimise dead time.

### Interferences

Interferences may originate from the sample or from the plasma or a combination of both. The interferences may be of polyatomic nature ( $\text{ArOH}^+$ ) or simpler ( $\text{UH}^+$ ) but all have in common that they interfere at mass position. The interferences may be avoided in different ways, either by removing the cause of them (eg removing the water also removes most hydrides and oxides) or by destroying them in collision cells filled with a gas of selected pressure and composition. They may also be avoided in high resolution MS by increasing the resolution. For the radioisotope work of U and Pu the most severe interferences are from hydrides since the  $\text{UH}^+$  cause a significant contribution at mass 239. This is frequently avoided by desolvating equipment in the sample introduction system to remove the water. Further problems may arise due to combination of Pb and Bi isotopes with chloride isotopes all having masses in the 230-240 range. The strong interferences caused at several masses by chlorides is the main reason why HCl seldom is used in ICP-MS samples.

### Abundance sensitivity (peak tailing)

A major limit in all ICP-MS instruments is the rather poor peak shape which means that the peak tail from major peaks disturb peaks of minor abundance. An example of how this may look is shown in figure 2. The strong tailing from the major isotope  $^{232}\text{Th}$  makes the determination of the minor  $^{230}\text{Th}$  very difficult. Unfortunately, the rather large spread in ion energy in ICP-instruments prevents the normal ion optics to produce better peaks. In high resolution instruments a combination of slits and energy filters may significantly improve the abundance sensitivity but the transmission is greatly reduced. Again, TIMS here shows a much better abundance sensitivity with some 0.01 ppm, compared to 5-50 ppm for ICP-MS, without compromising transmission.

The peak tailing is perhaps the largest problem in the analysis of extremely low levels of Pu-isotopes due to the presence of tailing from the  $^{238}\text{U}$ . For this reason the removal of uranium in the chemical separation procedure is of high priority and may ultimately determine to which level Pu may be measured. Without any separation it may be estimated that the detection limit for  $^{239}\text{Pu}$  in an ordinary 1g soil sample (containing  $20 \text{ mBq g}^{-1} \text{ }^{238}\text{U}$ ) is in the order of 100 mBq.

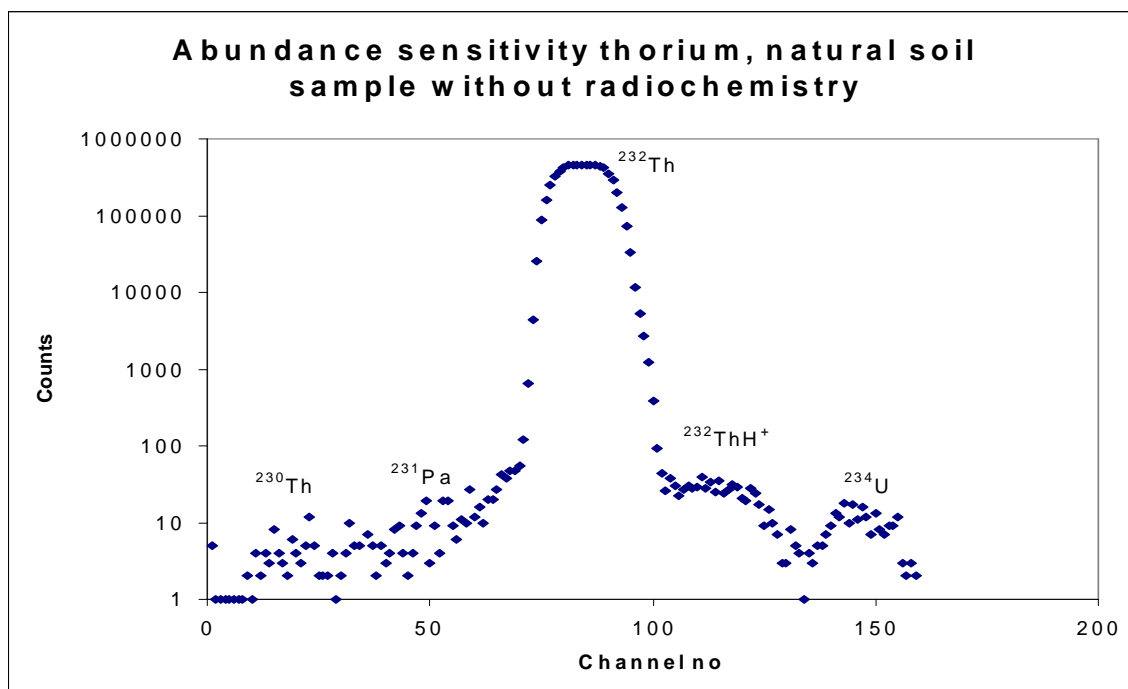


Figure 2:

Abundance sensitivity in low resolution mode (maximum transmission) for ICP-MS.

## Concluions

Although ICP-MS has made it considerably easier to produce MS data the need to oversee parameters governing precision and transmission has not decreased. The use of certified reference samples with well known isotopic ratios, suitable sample introduction systems and a well designed chemical separation is of great help in minimising at least the most severe problems in isotope ratio measurements.

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Key words                      Norcmass, ICP-MS, Mass spectrometry.